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HERMETIC COATING OF OPTICAL FIBERS

Spectran Corporation

S. Raychaudhuri, Philip S. Levin and Ray Sierra

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ROME AIR DEVELOPMENT CENTER
Air Force Systems Command
Griffiss Air Force Base, NY 13441-5700

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APPROVED: Osama El-Bayoumi

OSAMA H. EL-BAYOUMI
Project Engineer

Harold Roth

APPROVED: HAROLD ROTH
Director of Solid State Sciences

FOR THE COMMANDER: John A. Ritz

JOHN A RITZ
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Hermetically coated graded index silica fibers were to exhibit proof test levels greater than 200 Kpsi over one kilometer length Fiber was to exhibit a stress corrosion factor N greater than 100. The dielectric coating materials were to include, but not limited to diamond - like carbon and boron nitride. Techniques using hollow cathode discharge coater produced coated fiber with N value less than 26. Severe arcing problems were faced with this technique resulting in large cracks on the coater anodes. RF plasma coater was built and operated to deposit amorphous hydrogenated carbon (a - C:H) films both on bulk and fiber samples. Fibers were drawn under a number of conditions to determine the role, such factors as vacuum chamber alignment, tension, plasma conditions play in the strength of the fiber. A stress corrosion susceptibility factor N = 26 was obtained. In only preliminary experiments with the deposition of silicon nitride coating on the fiber, N values up to 70 were achieved. Keywords:

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SUMMARY

The objective of this program was to develop a low cost in-line process for producing multi-km lengths of hermetically coated fiber. The fiber was to be drawn from fused silica and silica based step index, and graded index preforms. The graded index, low loss silicate optical fibers, as drawn but without the hermetic coating, were to have less than 5 dB/km loss and bandwidth not less than 300 MHZ-km at 900 nanometers. The fibers were to be coated with carbon and boron nitride to establish and evaluate drawing and coating parameters. Hermetically coated fibers were to exhibit prooftest levels greater than 200 kpsi over one kilometer lengths. Fiber was to exhibit a static fatigue parameter N greater than 100. The dielectric coating materials were to include, but not be limited to, diamond-like carbon and boron nitride.

During Phase I of this program, a draw tower was assembled capable of drawing multikilometer lengths of multimode fiber. The draw furnace was modified to allow the draw process to be performed at reduced pressures. This eliminated the need for a differentially pumped entrance port to the coater, thereby reducing the risk of fiber damage prior to coating. The draw tower was outfitted with two polymeric coating stages. Each stage consisted of a pressurized hard die coating cup and a UV lamp for on-line curing of the polymer. The polymer buffers served to protect the bare fiber and hermetic coatings.

During Phase II, the initial effort was directed towards building a one meter hollow cathode discharge coater for coating the fiber. The coater section was isolated by means of differentially pumped stages from both the low pressure inert furnace invironment and from atmospheric contamination. The coater was operated and fiber drawn from silica rods was coated. Dynamic fatigue tests were performed with a gauge length of 11.5cm and a stress corrosion coefficient of N=26 was demonstrated. However, severe arcing problems were faced with this coater arrangement - resulting in large cracks on the coater anodes.

A decision was made at this stage of the program to implement an RF excited collisionally controlled glow discharge coater. This coater was operated to deposit amorphous hydrogenated carbon (a-C:H) films on both bulk and fiber samples. An optical multichannel analyzer was used to monitor the glow discharge in real time and optical emissions corresponding to efficient deposition were identified. Bulk NaCl, sapphire and glass samples coated with ~ 1 micron thick films were tested analytically. With the information gathered through bulk deposition experiments, efforts were directed to coat fibers with a-C:H films under dynamic conditions. Fibers were drawn under a number of conditions to determine the role such factors as vacuum chamber alignment, tension, plasma conditions play in the strength of the fiber. Modifications were implemented to allow better alignment of the exit ports.

Fibers coated with a-C:H were evaluated by dynamic fatigue testing. A stress corrosion susceptibility factor N=26 was obtained. To improve on the N value, experiments were conducted to coat fibers with materials like a-Si:H, a-SiC:H, Si_3N_4, TiC and BN. In preliminary experiments with the deposition of silicon nitride coating on the fiber, N values up to 70 were achieved, clearly verifying the capability of the RF plasma approach to produce reasonable hermeticity while avoiding the problem experienced with hollow cathode.

1.0 INTRODUCTION:

The surface of a freshly drawn glass fiber while seemingly smooth has many imperfections which when under stress, can grow and ultimately result in failure of the fiber. Moisture can greatly increase the growth rate of such flaws by chemical attack. Although typical communications fibers are coated with some type of polymer, such polymers provide little protection against moisture attack and the mean failure stress of the fiber decreases with time at essentially the same rate as an uncoated fiber. A thin hermetic coating would protect the surface of the fiber against moisture diffusion and would therefore preserve the initial strength of the fiber. Many such coatings have been investigated at SpecTran[5] and other facilities[1,2,3,4]. Ideally, a hermetic coating would be non-conducting and would

have no effect on the optical properties of the fiber. The coating would be applied during the draw phase of fiber manufacture and should be compatible with all other normally performed manufacturing steps (cabling, splicing, etc.).

The initial thrust of this program was to extend the development of a previously reported approach[6,7] for applying diamond-like carbon hermetic coatings on optical fibers. The technique involved the generation of a reactive glow discharge using a cylindrical-hollow magnetron coupled with a hollow cathode configuration. A mixture of methane and argon was introduced into the coater and flowed constantly during magnetron operation. This approach was not, however, successful due to severe arcing.

As a result, an RF excited reactive plasma deposition was adopted and more than ninety percent of the data generated during Phase II were obtained using this system. The remainder of this report describes in detail the work performed under this contract.

2.0 EXPERIMENTAL WORK AND RESULTS:

The thrust of this program was to combine the fiber draw technology with a viable hermetic coating process. Earlier work had identified the transition length from the exit of the draw furnace to the entrance of the hermetic coating

apparatus as a region subject to atmospheric contamination. In addition, the need for low pressure (< .1 torr) in the coater meant the unprotected fiber must enter the coater through a very small differentially pumped orifice. This latter requirement meant the fiber was exposed to potential damage due to surface contact with the orifice prior to coating. In order to alleviate both of these problems, the furnace was modified to permit draw at reduced pressures, and interfaced directly to the coater vacuum system. The resulting apparatus served to reduce contamination and provided an inert environment at the melt zone. In addition, since the size of the upper pumping port was greatly enlarged, alignment was less critical and the probability of fiber damage reduced. Fig. 1 is a diagram of the plasma coating chamber and associated vacuum system.

2.1 Cylindrical Magnetron Approach:

From November 1984 to December 1984, the cylindrical magnetron approach for coating fiber with a-C:H films was investigated. A long coater was designed and built to allow deposition at rates compatible with typical fiber draw speeds. The coater is shown schematically in Fig. 2. It consisted of a hollow cathode and two anodes coaxial with the fiber. Small (4mm) holes were drilled in the anodes which allow the fiber to enter and exit the coater section. The

total coater was 1 meter long with an inside diameter of 4.4 cm. The cathode section was constructed of high purity graphite and biased by means of Hippotronics high voltage D.C. power supply. An axial magnetic field between 80 and 300 gauss was applied with the intent of establishing magnetron oscillations.

The system as implemented was found to be subject to severe arcing near the anodes. Static experiments conducted using view ports confirmed the difficulty in sustaining a glow type discharge with the existing coater design. Although arcing prevented the coater from operating as intended, coatings were produced using constantly flowing mixtures of methane and argon. Both flow rates and compositions were varied. Silica rods drawn into fiber and coated by means of the discharge apparatus exhibited black sooty deposits. Fig. 3 shows the results of a compositional analysis performed on a typical coating using S.E.M/EDAX. As can be seen, the film consisted mainly of carbon. Hydrogen, a constituent commonly found in these films, was not detectable by S.E.M., but could not be ruled out. The resistivity of the coating was estimated from resistance measurements and found to be 10 Ω-cm. This low resistivity as opposed to the more insulating behavior from DLC implied the film was essentially impure graphite.

The coated fiber was tested using a dynamic fatigue tester (Instron) to determine static fatigue characteristics and hence evaluate coating hermeticity. Using a gauge length of 11.5cm, Weibull plots were generated at three strain rates covering two orders of magnitude. Stress corrosion co-efficients (N) were then calculated from the slope of a log-log plot of median failure stress vs loading rate[8]. As can be seen from Fig. 4, the data yielded a straight line. An N value of 26 was computed. This was in reasonable agreement with earlier work[6] where N=30 was reported. For comparison, Fig. 5 shows similar data for control silica fiber yielding a stress corrosion coefficient, N=21.2 indicating only slight improvement for the carbon coated fiber.

The hollow cathode design was carefully reviewed by both in-house personnel and discussed with Chatham Cooke, a consultant from MIT. It was concluded that large aspect ratio (length of cathode divided by inside diameters) led to a very unstable discharge. The reason for this instability was the large voltage required to initiate a very low pressure (< 0.1 Torr) discharge. It is often the case that at these large values of E/N (electric field divided by gas number density), secondary emission by ion bombardment of the electrodes

becomes the dominant source of electrons. Localized hot spots can develop and arcing and sputtering commonly occur. As a result, the coatings deposited using the hollow cathode apparatus were found to be essentially graphite and provided only very slight improvement in static fatigue parameters.

2.2 Reactive RF Plasma Approach:

Collisionally controlled plasma glow discharges can be used to deposit varieties of thin film coatings[9,10] including a-C:H. Such discharges are characterized by operation in a pressure region where collisional processes in the bulk gas control both the production and loss of electrons and consequently determine all discharge parameters. In such discharges, the role of the electrodes is minimized. Owing to the efficiency with which collisional processes can distribute energy, the mean kinetic energies of both the neutral and ionic species tend to reach a thermal equilibrium. This is in marked contrast with the low pressure (< 100 mTorr) electrode controlled glow discharge commonly used in magnetron deposition work. Thus, the problem of deposition from a collisionally controlled glow discharge reduces to the creation, by means of gas parameters (pressure, composition, etc.), of a chemical environment that supports the deposition of a-C:H.

One convenient way of generating a collisionally controlled plasma glow discharge is by means of a low power RF source. Most gases in the pressure range from 0.1 Torr to 10 Torr are readily ionized by alternating fields in the 1-30 MHZ frequency range. A number of features of RF excitation make it an ideal candidate for use here. First, since the power supply can be inductively coupled to the coater, the need for electrodes (and consequently the feed-throughs, etc. required for these) disappears and vacuum integrity can be improved. The length of the coating region can be made arbitrary since any number of coupling coils can be utilized. Finally, the system affords a means of varying conditions easily.

The hollow cathode coater was replaced with a silica glass tube of the same length and sealed by means of O-ring compression fittings. The tube was fitted with two ports, one for the introduction of the reacting gases, the other for pressure monitoring. A block diagram of the overall system is given in Fig. 6. Two low power (~ 150 watts) RF generators (marked RF 1 and 2) were used to excite the gases within the coater. The generators used were ordinary ham radio power supplies operated in the 14 MHZ band. A commercial matching unit was used with one of the transmitters allowing nearly

100% coupling to the coater while a second matching unit was constructed with available parts in-house and typically provided 60% coupling to the coater. Emissions from the discharge were monitored in real time using the O.M.A. (optical multichannel analyzer). The O.M.A. used was a P.A.R. 1452 plasma monitor consisting of a 512 element Reticon scanned array mounted on a Y.A. 0.25 meter monochromator. The overall spectral resolution of the system was on the order of 0.5 nm. Because no pressure gauge was available at the coater, it was impossible to know the actual pressure in the coater. Instead, flow rates were measured and the pressure assumed to vary proportionately.

Several gas combinations were used. These were various mixtures of butane/He, butane/Ar, acetylene/He, freon/He and pure butane and acetylene. A typical spectrum taken from a butane/He discharge is presented in Fig. 7. This spectrum is typical of the conditions that were found to lead to high deposition rates. Basically, it consisted of well developed band sequences extending over the entire visible region of the spectrum. It was found that deposition rate increased with increasing He/Butane ratio. Atomic hydrogen lines were prominent in the spectra of these high deposition mixtures. The vast majority of the bands remain unidentified however.

Qualitatively, these results are easily understood. Electrons are produced by impact ionization of the gas. In the case of He, the process may be expressed:

$$He + e^- \rightarrow He^+ + 2e^- \quad --(1)$$

For the butane, we have a number of possible steps:

$$\begin{aligned} C_4H_{10} + e^- &\rightarrow C_4H_9^+ + 2e^- \\ &\rightarrow C_4H_8 + H^+ + 2e^- \quad --(2) \\ &\rightarrow C_4H_8^+ + H + 2e^- \\ &\rightarrow \text{other fragments} + 2e^- \end{aligned}$$

As the partial pressure of butane is dropped, the probability of the ionization becomes dominant. In this case then, the types of fragments produced are determined by the results of collisions of the type

$$He^+ + C_4H_{10} \rightarrow He + \text{fragments}^+ \quad --(3)$$

Since the ionization potential of He is quite large (> 580 kcal/mole), the results from (3) can be rich in highly reactive free radical fragments which can then undergo reactions at any surface they may encounter.

Thus, the main feature of the data could be

explained. As the partial pressure of butane was decreased, impact ionization of the helium (equation 1) became dominant and subsequent ion-molecule reactions (equation 3) became the principal mechanism of formation of butane fragments. Among these fragments was atomic hydrogen which accounted for the observed Balmer lines. The deposition rate increased due to abundance of reactive fragments. Deposition from both helium-butane and argon-butane mixtures showed the same behavior. The best deposition rate was obtained with a 1% helium-butane mixture which gave 400 Å/min.

Deposition from acetylene was remarkably different. The spectrum of a pure acetylene discharge is shown in Fig. 8. As is evident here, the spectrum shows a strong atomic hydrogen component. Furthermore, a sequence of bands appear to the blue of the H-Balmer α-line which have the appearance of a predissociation spectrum. All efforts to identify these bands from the literature failed to provide positive identification. The deposition rate was found to be largest for acetylene discharges at relatively low pressures. The highest rate was found to be of order of 450Å/min.

A number of bulk samples were coated with a-C:H including microscope slides, NaCl plates, ZBLAN fluoride glass and sapphire blanks. IR transmission spectra was

collected in many of these cases. Table I gives a list of the gases and gas mixtures used along with the observed deposition rates and appropriate comments. A number of conclusions could be drawn from these experiments.

It was evident that a-C:H films could be deposited from collisionally controlled plasma glow discharges. Helium was a better buffer gas than argon and led to higher deposition rates. It was not clear whether this was due to better coupling of the RF in helium or due to more efficient ion-molecule reactions. Similarly, butane provided higher deposition rates than did methane.

Attempts to use fluorinated compounds were not successful, although it is not clear why this should be the case. The fluorinated species are strongly electronegative and no doubt form negative ions. Whether they were in some way inhibiting a-C:H formation would be total speculation at this stage.

Finally, the acetylene discharge was interesting. Clearly significant fragmentation (as evidenced by both atomic hydrogen lines and the apparent predissociation bands) was taking place independent of buffer gas.

Analyses of the films were made in several different ways. For sufficiently thick films, the SEM could be used to determine both film thickness and composition. In the best case, only C, Si and O could be identified. Fig. 9 shows the spectrum of a film deposited from 90:10 He: Butane gas mixture. In other cases, a number of trace impurities such as Na, Mg, Al, etc. were detected but these appear to be part of the substrate material. The SEM could provide no information on the structure of the films and was unable to detect atomic hydrogen which could be as much as 30 at. % of the film. In all cases, the films were non-conducting (as determined using an ohm meter) and had a brown tint to them.

Infrared transmission spectra in the 2-10μm range were obtained using an FTIR spectrometer. The spectra were found to be similar to that reported by others. A typical spectrum is shown in Fig. 10. The bands near 3.5μm are due to C-H stretch and have been observed in a-C:H films produced by other means of deposition. The bands between 6μm and 10μm are mainly due to impurities except that near 7.4μm, it may be C-C stretch. Figure 11 shows the C-H band expanded. Bubenzer et.al. analyzed the structure of this band and obtained the relative contributions of sp^2 (graphite-like) and sp^3 (diamond-like) bonding in their film. A similar

analysis of film deposited at SpecTran indicates ~ 50% sp^3 character. A typical transmission spectrum derived from a film deposited from pure acetylene discharge is shown in Fig. 12. Aside from the bands near 2900 cm^{-1} a weak band was observed at 330 cm^{-1} corresponding to C-H stretch on sp^1 bonded carbon (carbyne like). This spectrum is very similar to what has been observed by Bubenzer et.al.[11] using benzene in a self biased discharge.

Resistance to various solvents and to HF had been tested with a number of these films. In some cases, for example, for a film deposited from an acetylene discharge on a sapphire substrate, adhesion proved to be quite good. For these cases, films easily passed an adhesive tape test, could be cleaned with solvents, and rubbed vigorously with lab tissue. It was often the case however that thick films would not show such adhesion. It was also found that HF would not wet the surface of the films and had no observable effect on the films even after several hours. It was observed, however, that after ~ 1 hour of HF exposure, the film would lift in some places. This was probably due to the existence of pinholes in the film through which the HF penetrated and attacked the substrate underneath.

During the period March 1985 to September 1985,

efforts were directed towards coating fibers with a-C:H films with the plasma coater used for bulk deposition experiments. Initial attempts to draw the fiber with the vacuum system and coater fully assembled yielded very weak fiber. The fiber when stressed would break in many pieces. Inspection of the glass under a microscope revealed severe scratches caused by contact with one or more of the 4mm differential pumping ports. Alignment of these ports had been critical to fiber draw and view ports had been added at several points to permit careful alignment. Inspection of one of the sections not fitted with a view port revealed alignment problems which, when corrected, immediately led to drawing good strength fiber. Figure 13 shows a typical Weibull plot generated from data on tensile testing of uncoated control fibers. Median strengths were in the range of 700-750 kpsi. The plasma coater was then operated to deposit thin films of a-C:H on the fiber under dynamic conditions. Draw speed was typically between 5-10 meter/min. The vacuum was maintained between 0.1 to 0.3 Torr. An He: acetylene mixture was used. For all cases the fiber was visibly coated with a light brown deposit.

Analysis of the fiber proceeded as follows. A number of breaks were made using a 0.5 meter gauge length fiber in the Instron dynamic fatigue tester. After ~ 20 breaks at each of two strain rates two orders

of magnitude apart, a stress corrosion coefficient was evaluated. Because the break distribution was quite wide, the values of the stress corrosion coefficient showed a wide variation. But the best estimate of N value with a-C:H coatings was 26, which showed that a-C:H were ineffective so far as hermeticity was concerned. Figure 14 presents the Weibull plots of a-C:H coated fibers tensile tested at two different strain rates. The reason for this lack of hermeticity may be the columnar structure of these coatings. A scanning electron photomicrograph on a-C:H coating clearly shows the columnar nature of film growth. Figure 15 shows a typical example.

To improve upon the moisture barrier properties further, a few other materials were explored as potential hermetic coatings.

Amorphous Si:H coatings were deposited on the fiber using the plasma coater. Gaseous silane was used as the precursor material instead of a hydrocarbon. The coating was quite transparent in the infrared wavelengths and had a refractive index of approximately 4.0. It was a good insulator but provided no hermetic protection.

Attempts were made to deposit silicon carbide,

titanium carbide, silicon nitride and boron nitride on fibers by the plasma technique. For silicon carbide, a mixture of silane and acetylene was fed into the plasma coater. A hard coating was obtained and an N value of as high as 60 resulted. Figure 16 presents the data in the form of Weibull plots.

For silicon nitride deposition, a mixture of silane and nitrogen was used. Fiber drawn at up to 10 meters/minute showed hermetic protection with a stress corrosion coefficient, N=75. Results are shown in Fig. 17.

Initial work with titanium carbide deposition from a mixture of $TiCl_4$ and acetylene led to serious plasma instability problems. Black sooty deposit was obtained on the fiber. Boron nitride could be easily deposited from a mixture of diborane and nitrogen. The diborane was diluted in hydrogen and, therefore, no inert carrier gas was required. While BN did not show any hermetic protection (N=18), it seemed to provide the fiber with a scratch resistant surface.

3.0 CONCLUSIONS:

Initial efforts in this contract were directed towards coating of fiber with a-C:H by the hollow

cathode magnetron approach. One such coater was designed and implemented on a draw tower. But it was found that the coater was subject to severe arcing. The electrodes were rapidly eroded. Fiber was essentially found to be coated with impure graphite with only very slight improvement in static fatigue parameter.

After careful review of the coater design, it was concluded that the large aspect ratio (length of cathode divided by inside diameter) of the coater led to discharge instability. A solution to this problem might have been the use of a series of small L/D ratio coaters. Since each of these coaters would have had to be operated under low vacuum, the fiber would have had to pass through that many more entry and exit vacuum ports. This would have increased the problem of aligning the system and all the ports - a critical necessity for making fibers with high strength.

To address some of these problems and to make the deposition system easily operable and maintainable, the hollow cathode apparatus was replaced by a low pressure RF plasma coater. Collisionally controlled plasma discharges in hydrocarbons were successfully used to deposit a-C:H films. There was no need for biasing the substrate or in any way controlling the ion energy to obtain films with desirable properties. The coater used

in these experiments was far simpler than the hollow cathode device it replaced and certainly much cheaper to maintain. Deposition rates as high as several angstroms per second were achieved.

Much of the effort was dedicated towards drawing optical fiber with this a-C:H coating in a continuous fashion. Fibers could be successfully coated with diamond-like carbon. But the coating did not provide the necessary hermeticity as was seen from the low N value. The reason for this may be the columnar structure of these coatings. A possible solution to this problem may be to increase the coating thickness by extending the plasma coater length. Height limitations at the time of this contract precluded extension of the coater length.

Several other materials besides a-C:H were evaluated as potential hermetic coatings. These were a-Si:H, silicon carbide, silicon nitride, titanium carbide and boron nitride. Both the carbides and silicon nitride showed promise as potential hermetic coatings for fiber by the plasma approach. N values as high as 70 were obtained with silicon nitride.

Further investigations with materials like silicon nitride, silicon carbide or titanium carbide were

indicated by this work. These materials appeared to be promising as potential hermetic coatings for optical fibers. The work done during this contract also demonstrated the viability of adopting the RF plasma deposition as a process easily compatible with the conventional optical fiber draw technology.

Although the original hollow cathode discharge technique failed to produce diamond-like carbon and was abandoned in favor of the RF excitation approach, and even though the RF excitation approach could produce diamond-like carbon with only low N value, the program did demonstrate:

(a) the feasibility of direct interfacing of a draw furnace and vacuum coater;

(b) the feasibility of increasing the sizes of entrance and exit orifices of the deposition chamber to produce undamaged control and coated fiber;

(c) the feasibility of using RF discharge in producing diamond-like carbon;

(d) the applicability of RF discharge technique to several chemistries capable of providing hermeticity;

(e) possibility of using RF discharge technique to apply hermetic coatings at low temperatures;

and (f) potential use of RF deposition technique to apply hermetic coatings on an important class of glass optical fibers - heavy metal fluorides.

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LIST OF TABLES AND FIGURES

TABLE I

SUMMARY OF DEPOSITION EXPERIMENTS

SUBSTRATE	GAS COMPOSITION		DEPOSITION RATE	COMMENTS
NaCl	Ar/Butane		1 μm/hr	
	He/Butane	18:1	250 A/min	poor adhesion
		45:1	330 A/min	
		90:1	415 A/min	
	Acetylene		450 A/min	
	He/Methane		low	
	He/CCl_2F_2		negligible	
Sapphire	Acetylene			good adhesion
Glass slides	He/Butane			good adhesion
	Acetylene			shows some acid resistance
ZBLAN	He/Butane			shows some water resistance
Fiber	He/Butane		not observable	shows some acid resistance

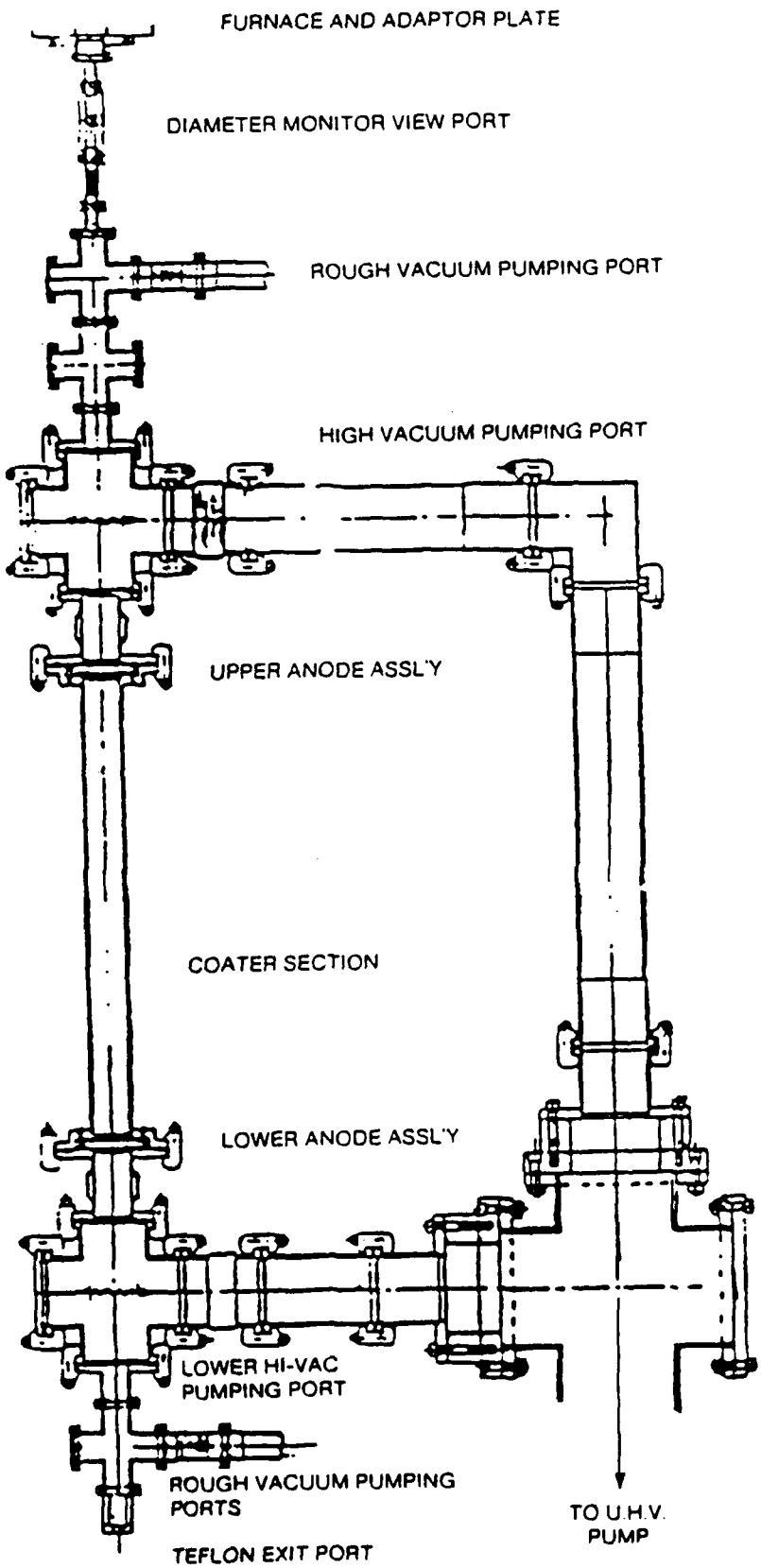

FIGURE 1.

Overall View of the Hermetic Coating Apparatus

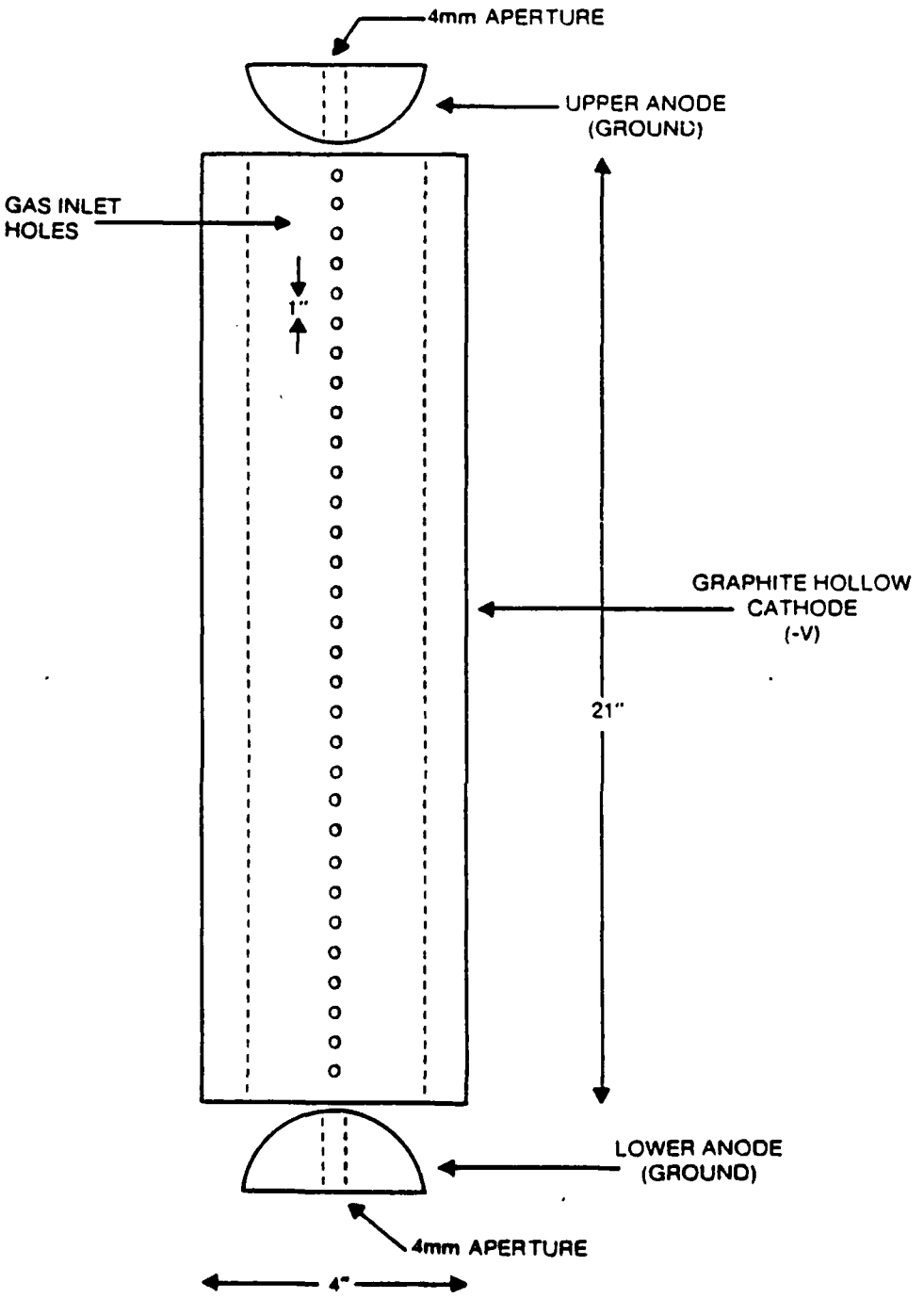

FIGURE 2.

SCHEMATIC DIAGRAM OF THE COATER

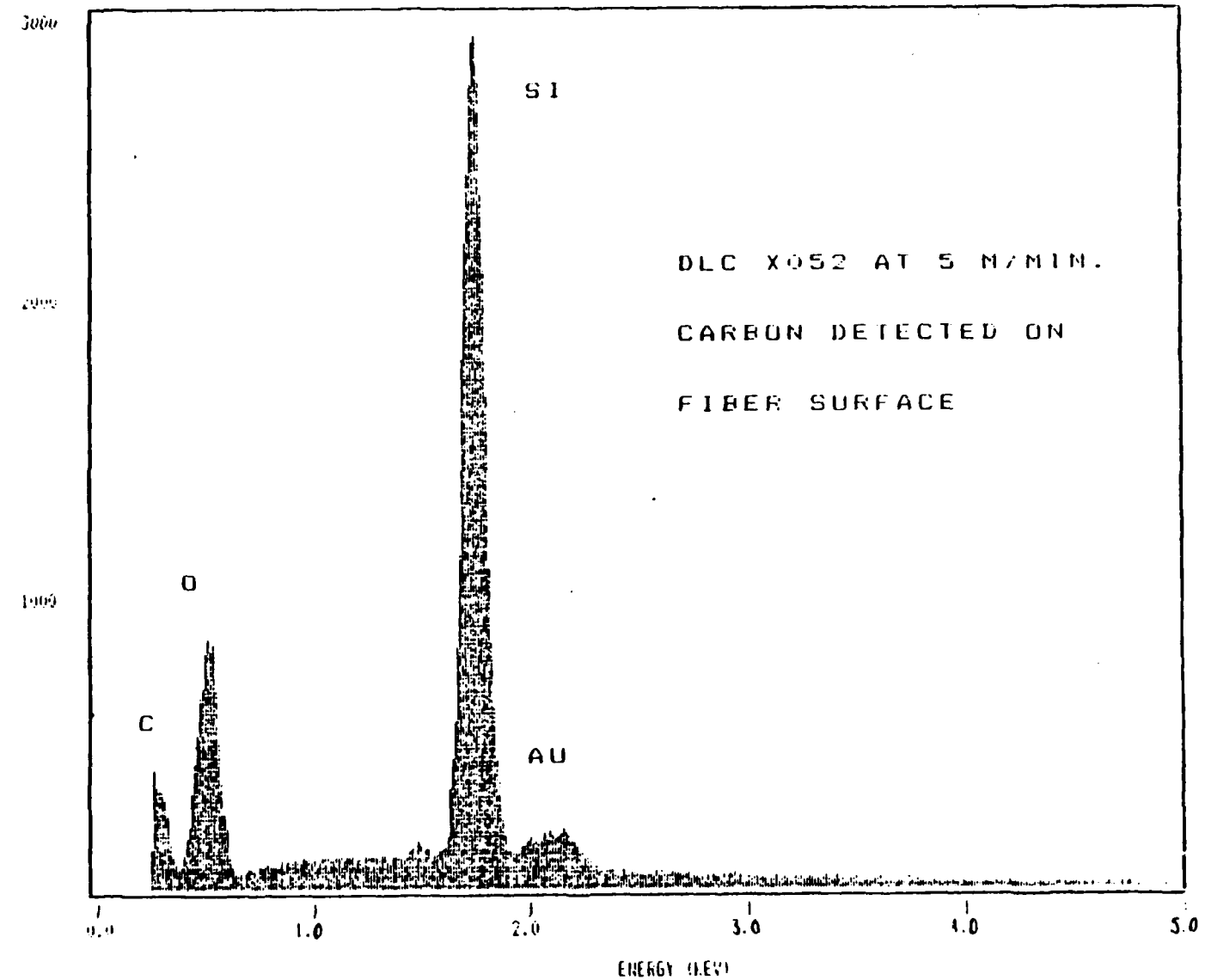

FIGURE 3

FIBER COATING ANALYSIS

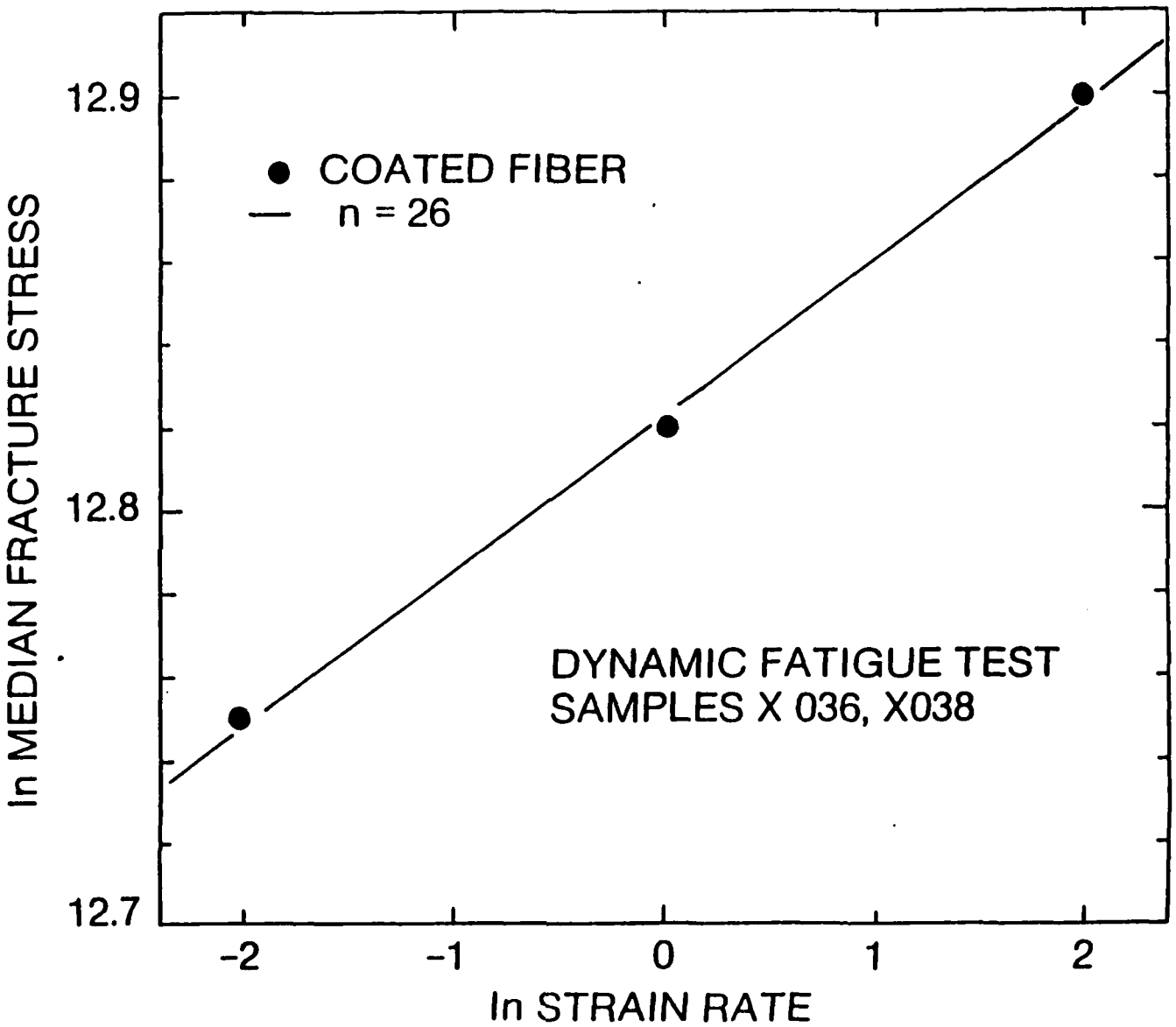

FIGURE 4.

DYNAMIC FATIGUE PLOT - COATED FIBER

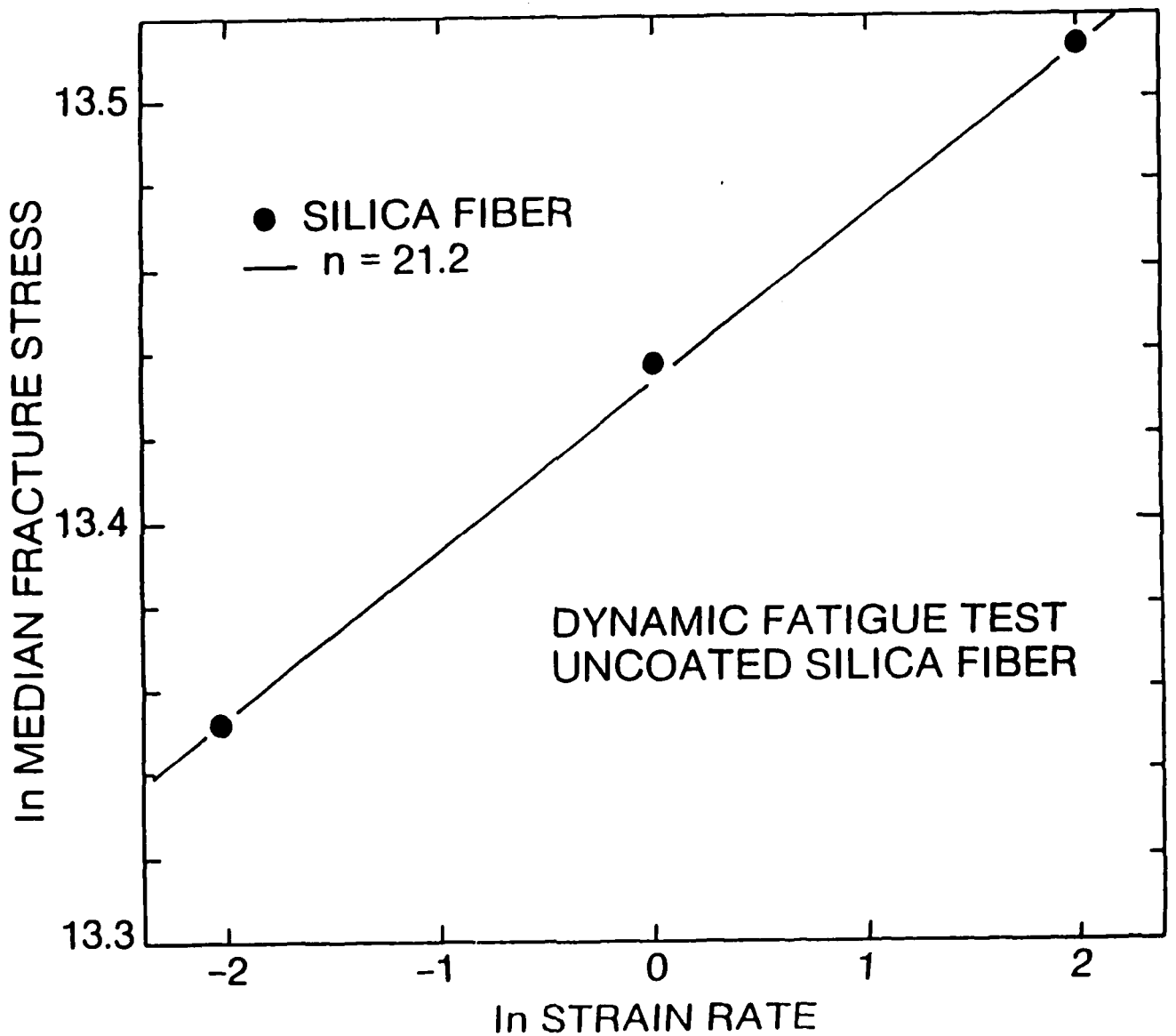

FIGURE 5.

DYNAMIC FATIGUE PLOT - UNCOATED FIBER

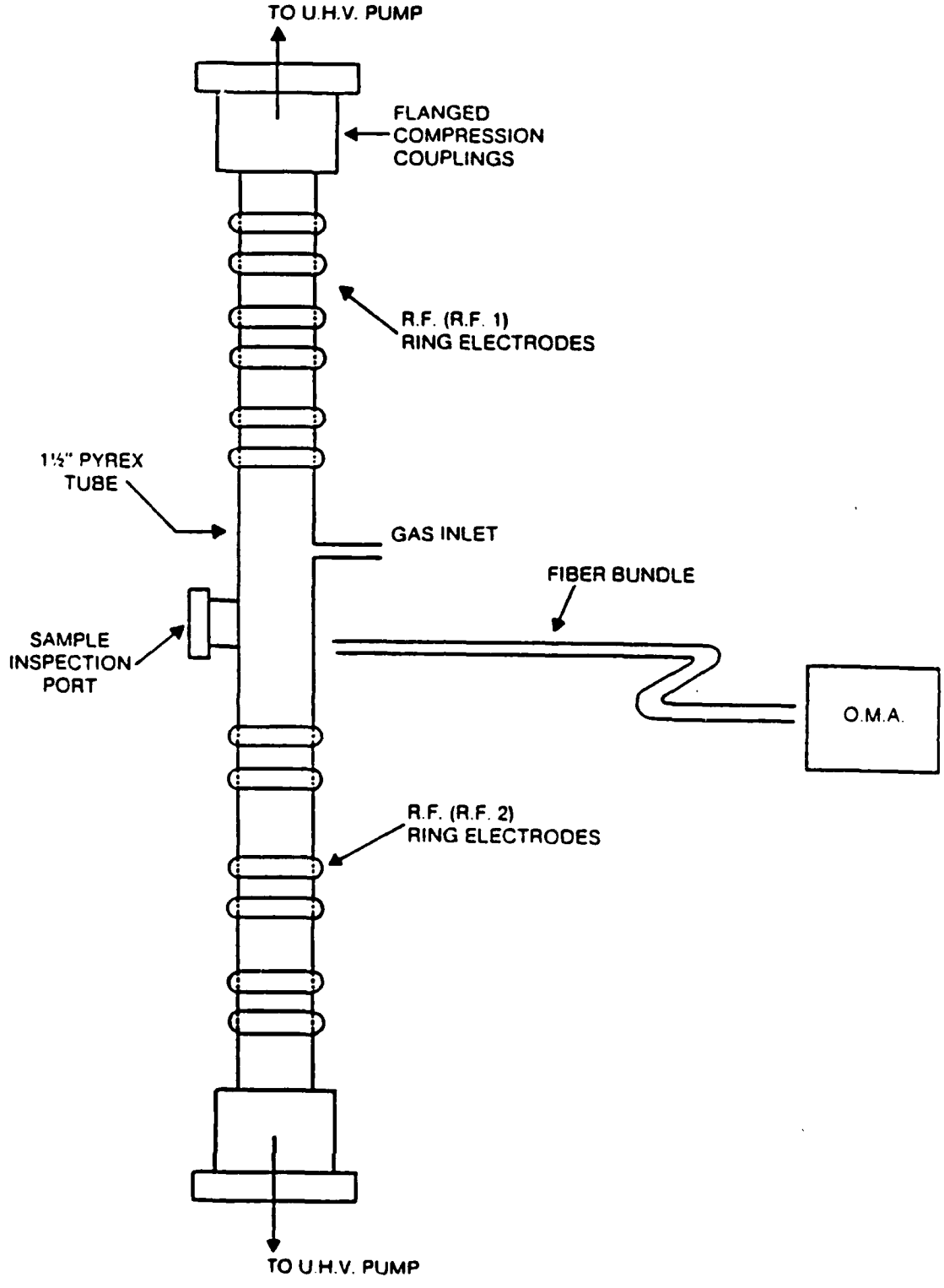

FIGURE 6.

SCHEMATIC DIAGRAM OF PLASMA COATER

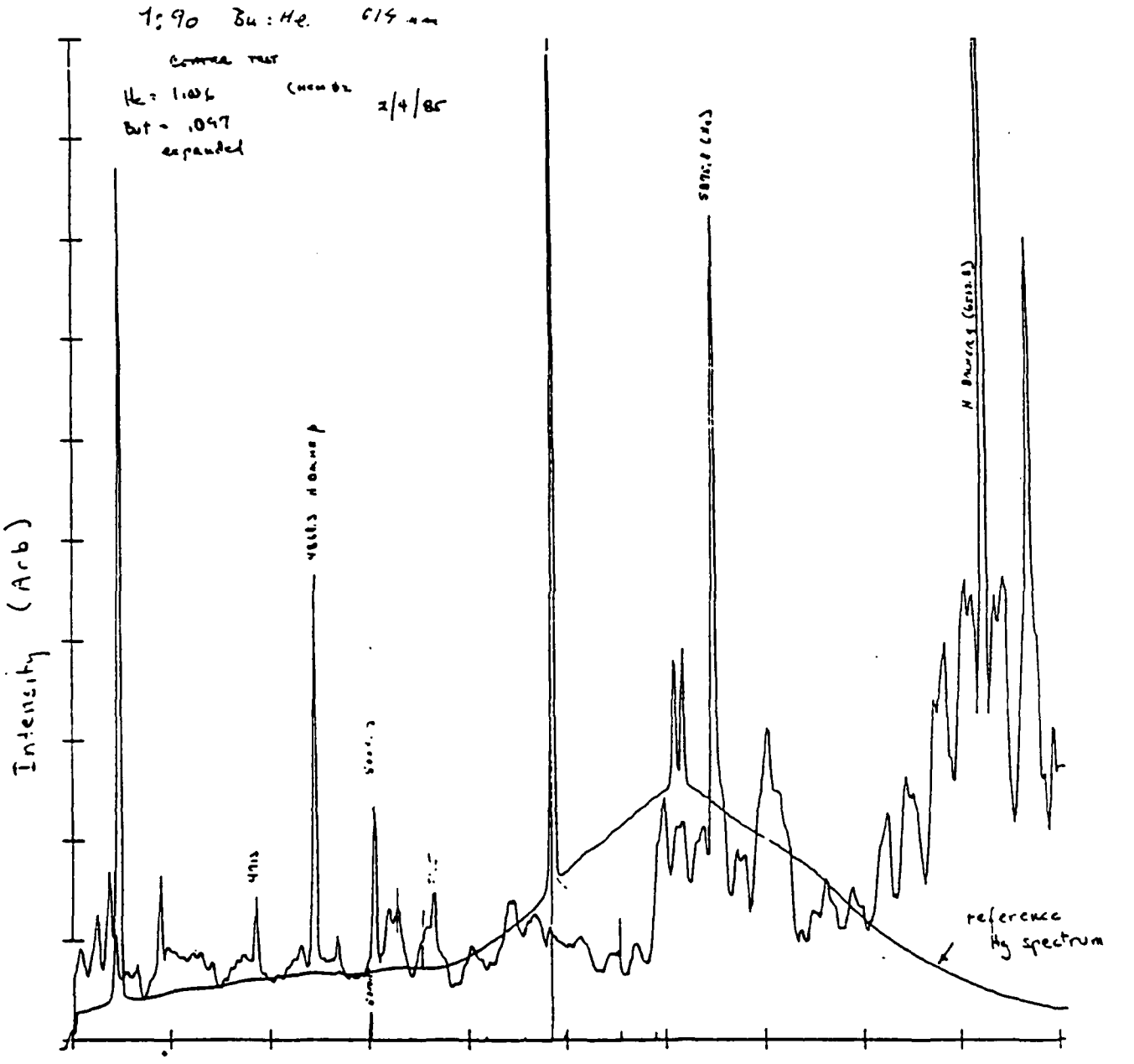

FIGURE 7.

OPTICAL EMISSION SPECTRA - He/BUTANE MIXTURE

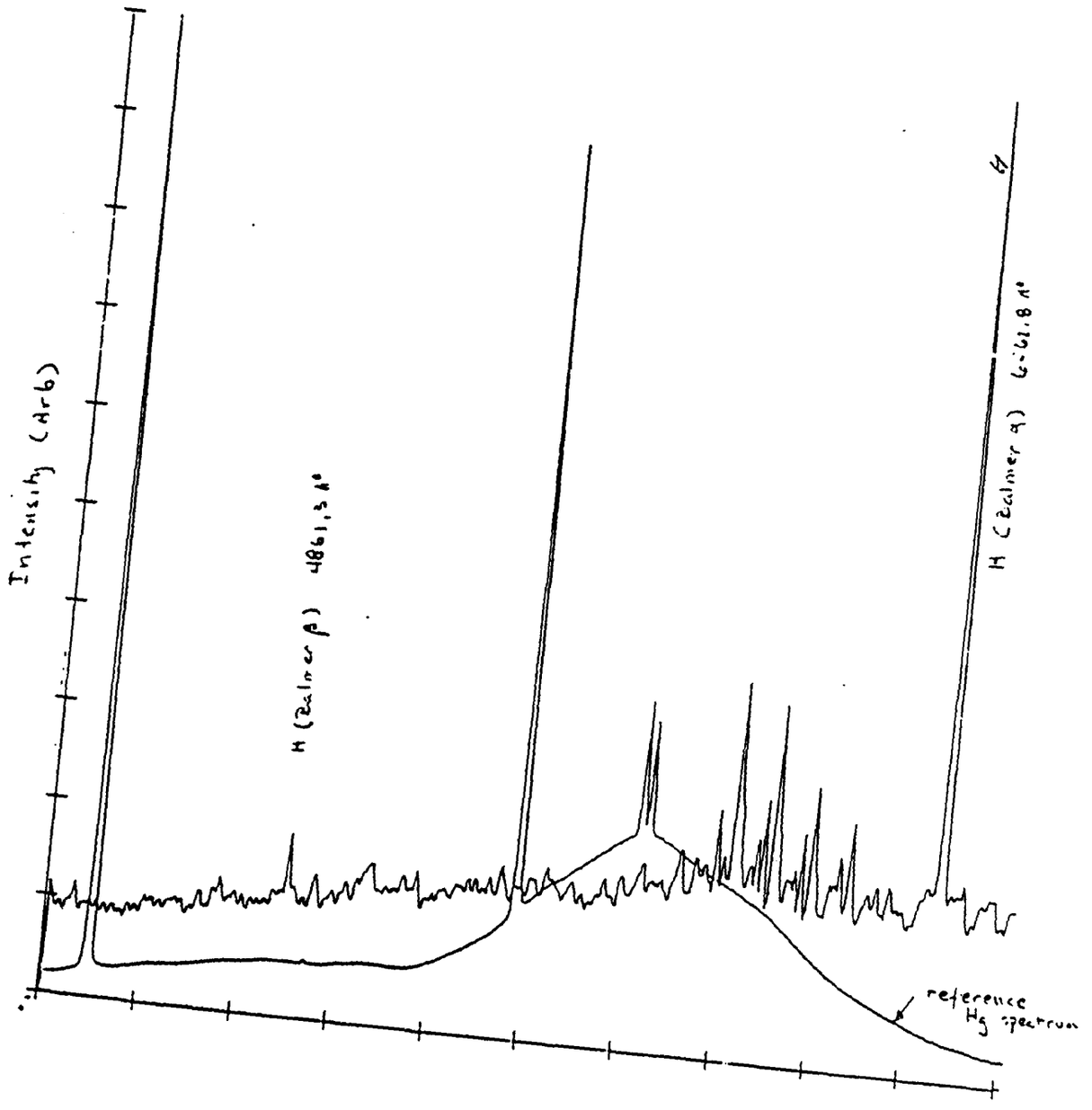

FIGURE 8.

OPTICAL EMISSION SPECTRA - ACETYLENE DISCHARGE

CARBON FILM ON GLASS SLIDE #4

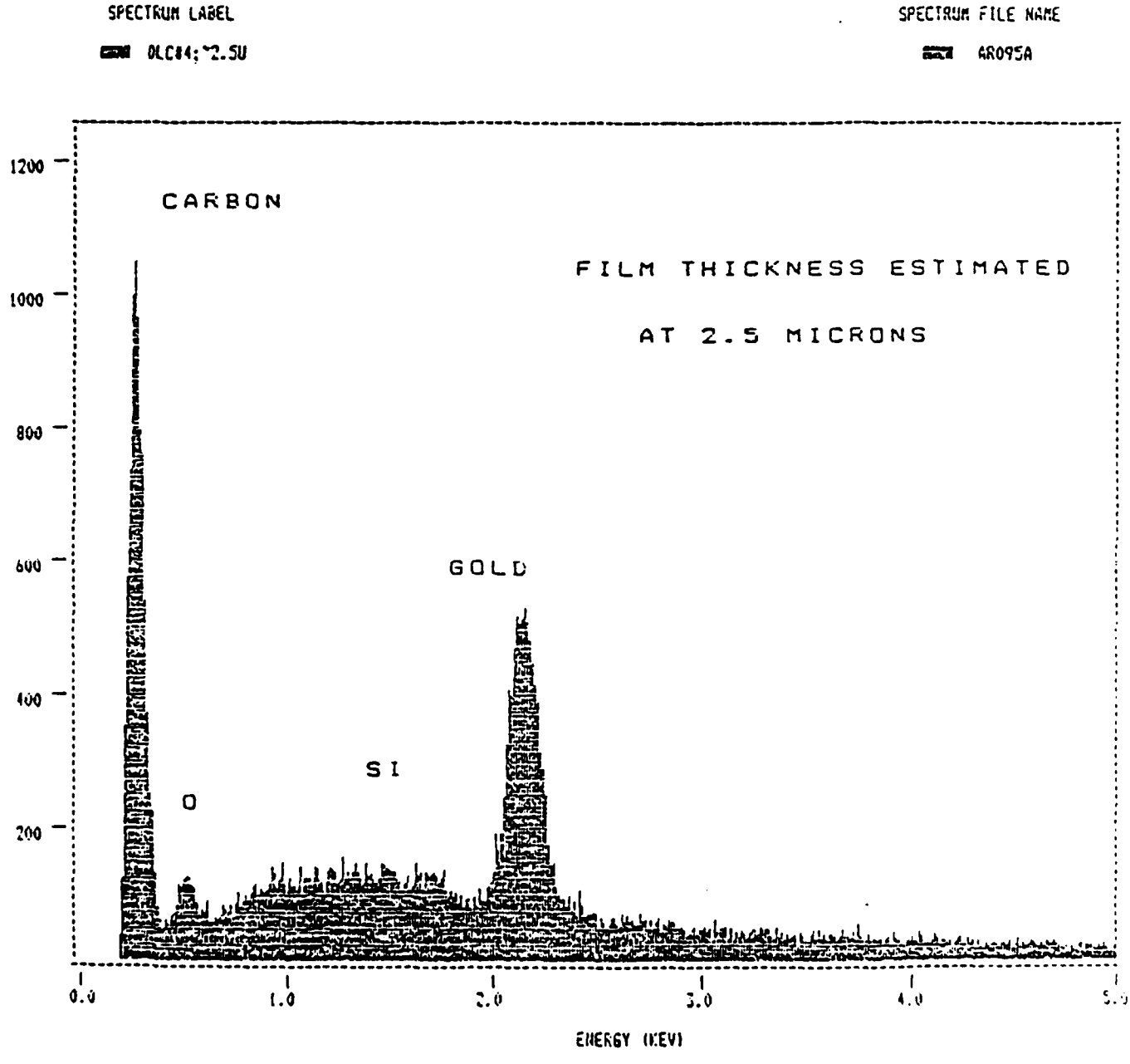

FIGURE 9.

SEM ANALYSIS OF CARBON FILM

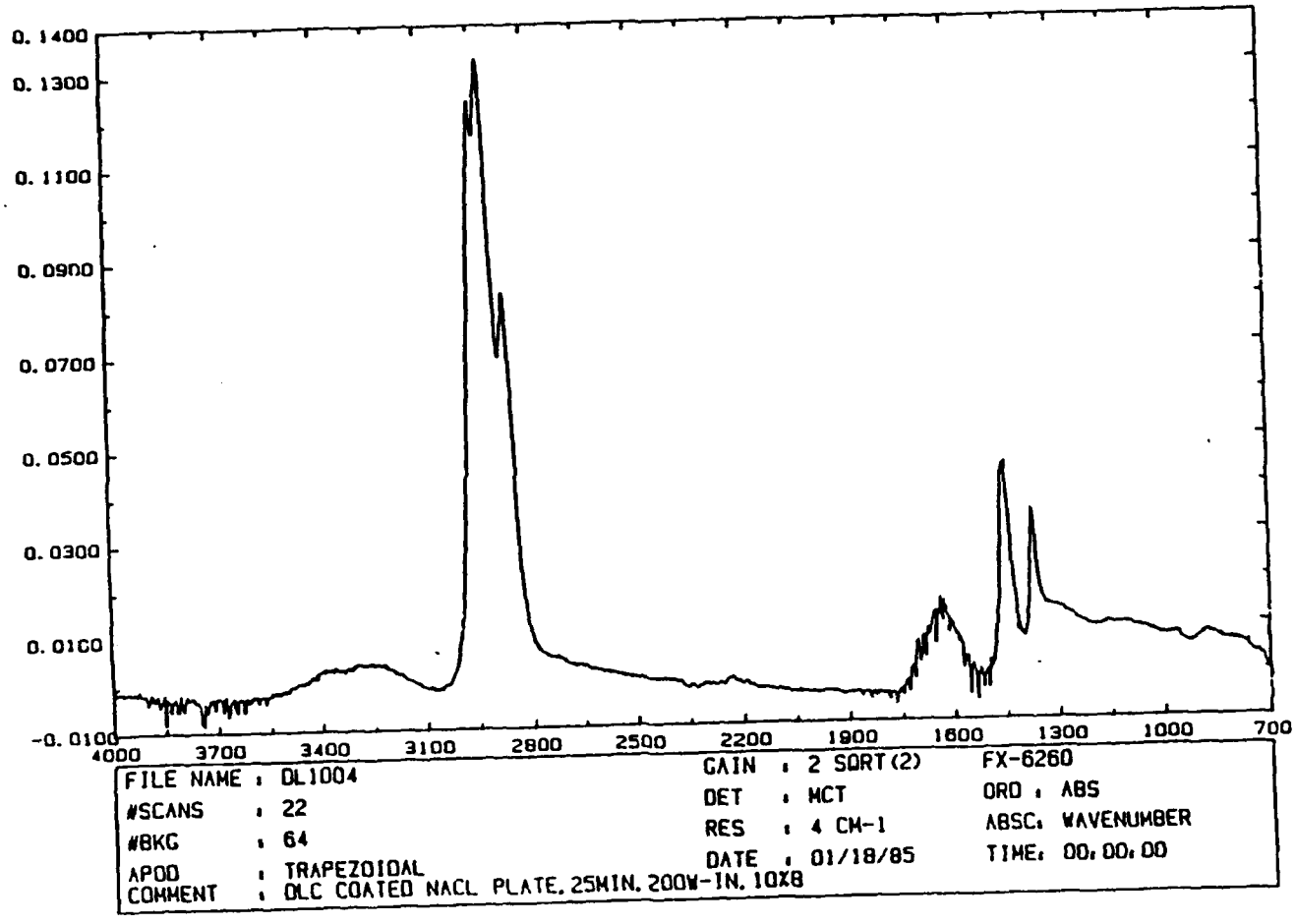

FIGURE 10.

IR SPECTRA a-C:H FILM

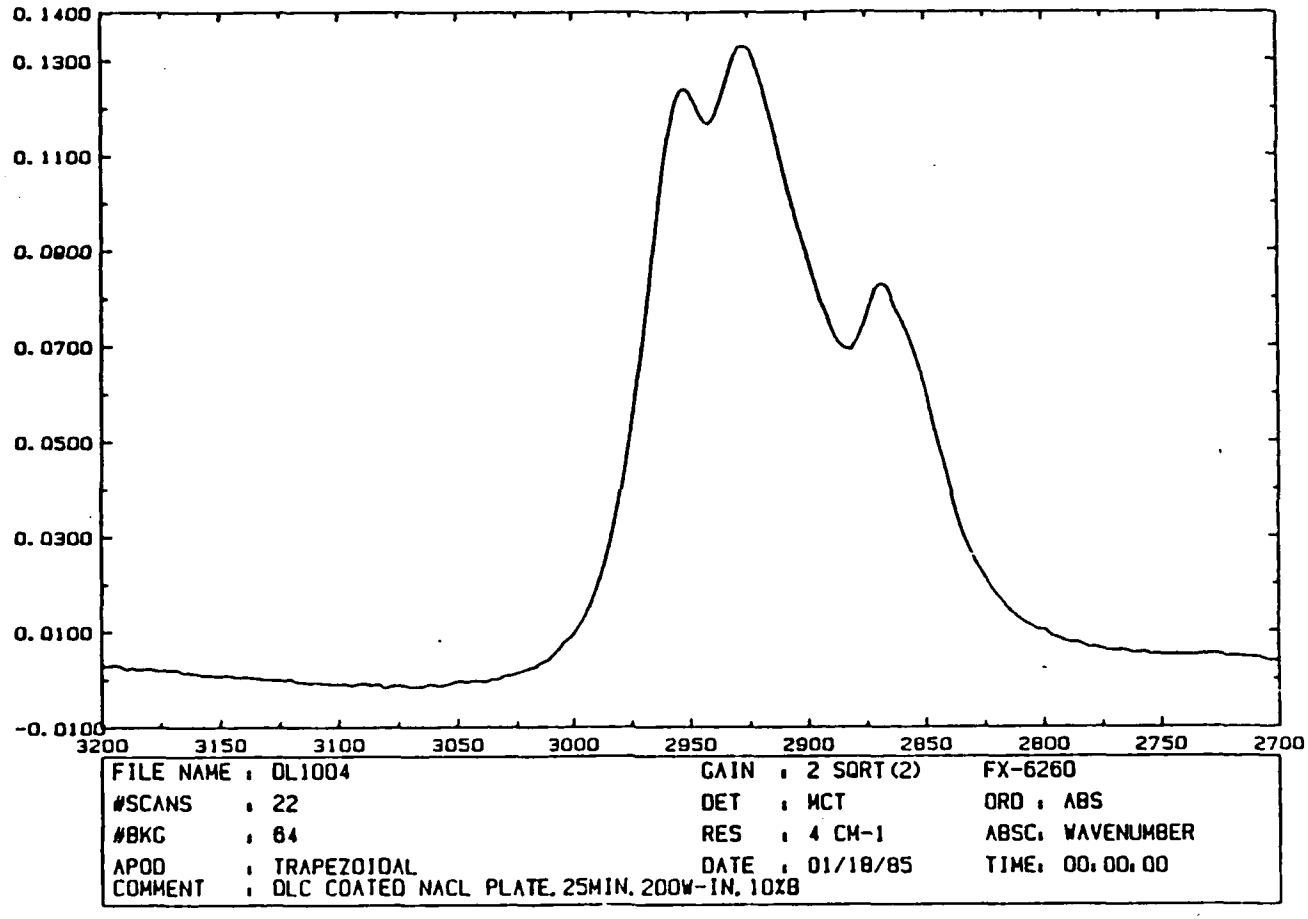

FIGURE 11.

C-H BAND STRETCH - IR SPECTRA

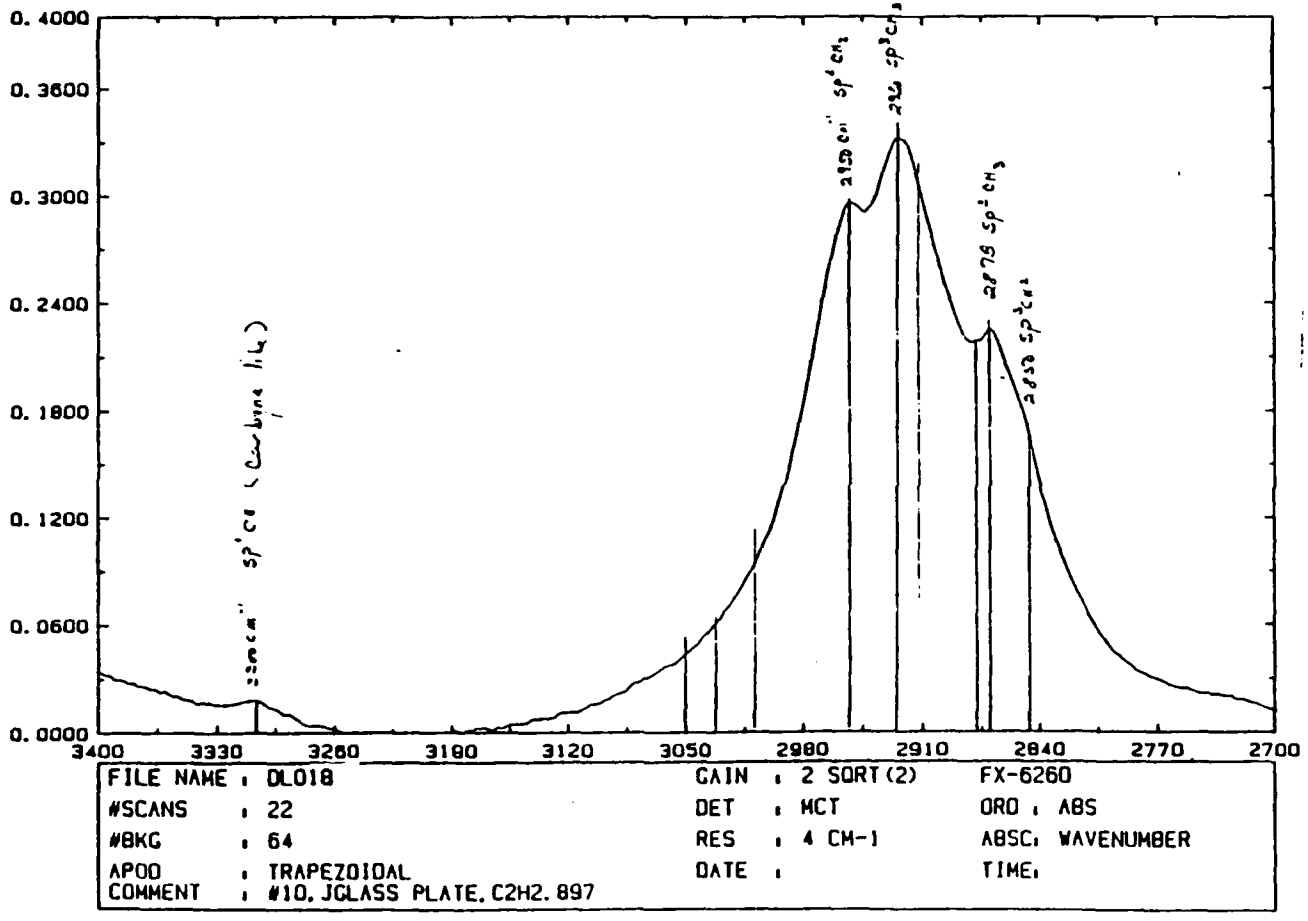

FIGURE 12.

IR SPECTRA OF a-C:H FILM FROM ACETYLENE DISCHARGE

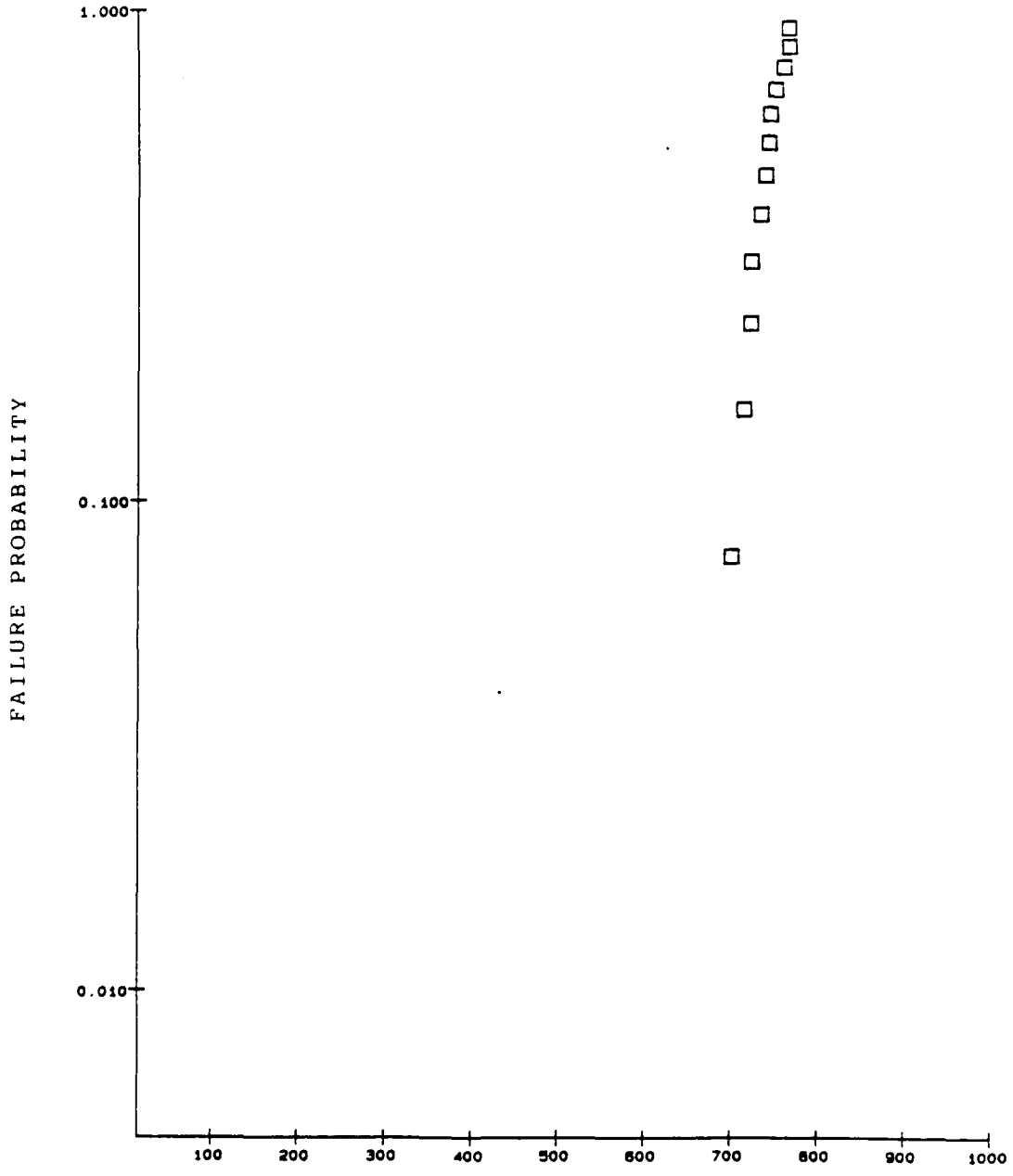

FIGURE 13.

WEIBULL PLOT OF CONTROL FIBER

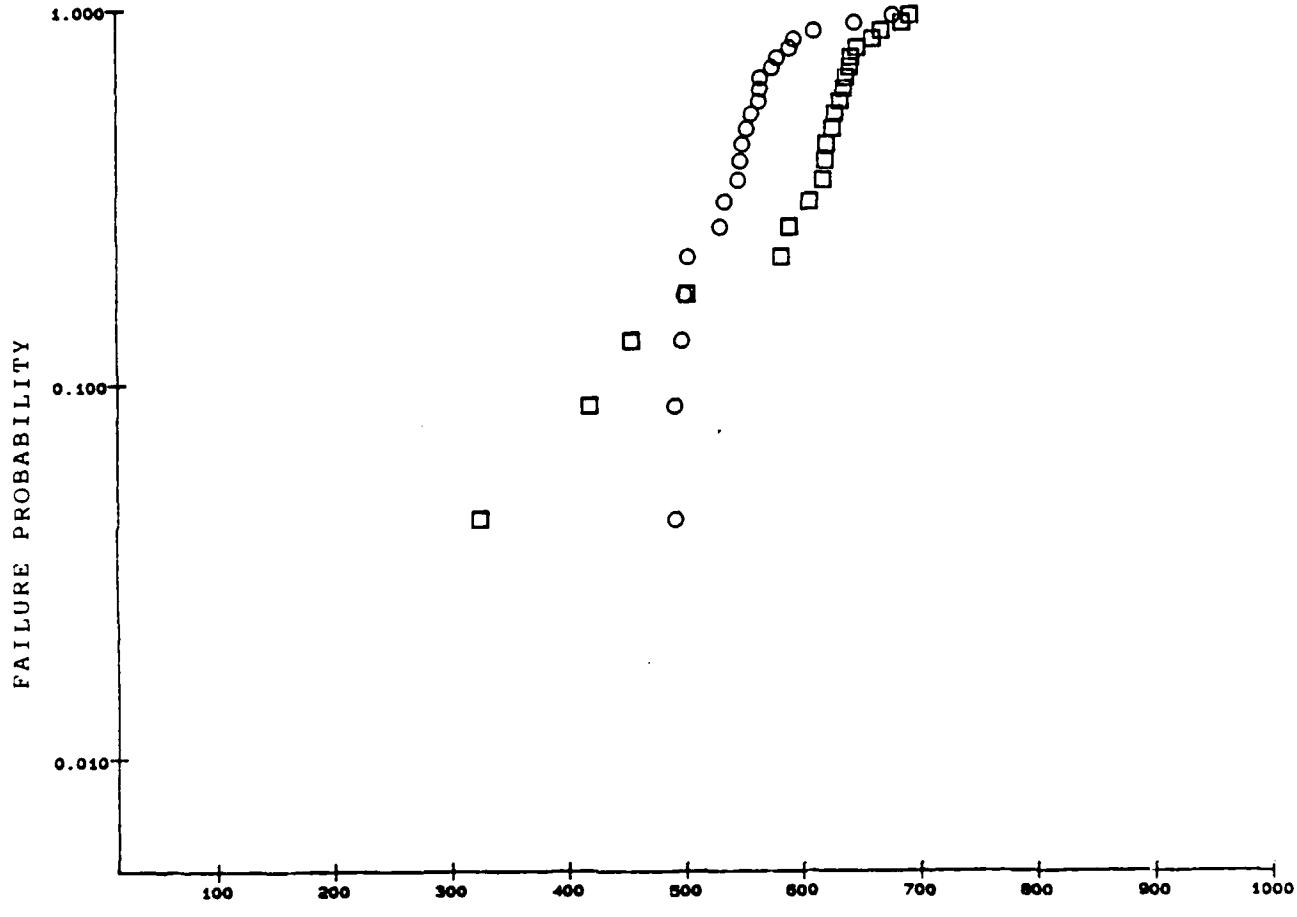

FIGURE 16.

WEIBULL PLOTS OF SiC COATED FIBERS

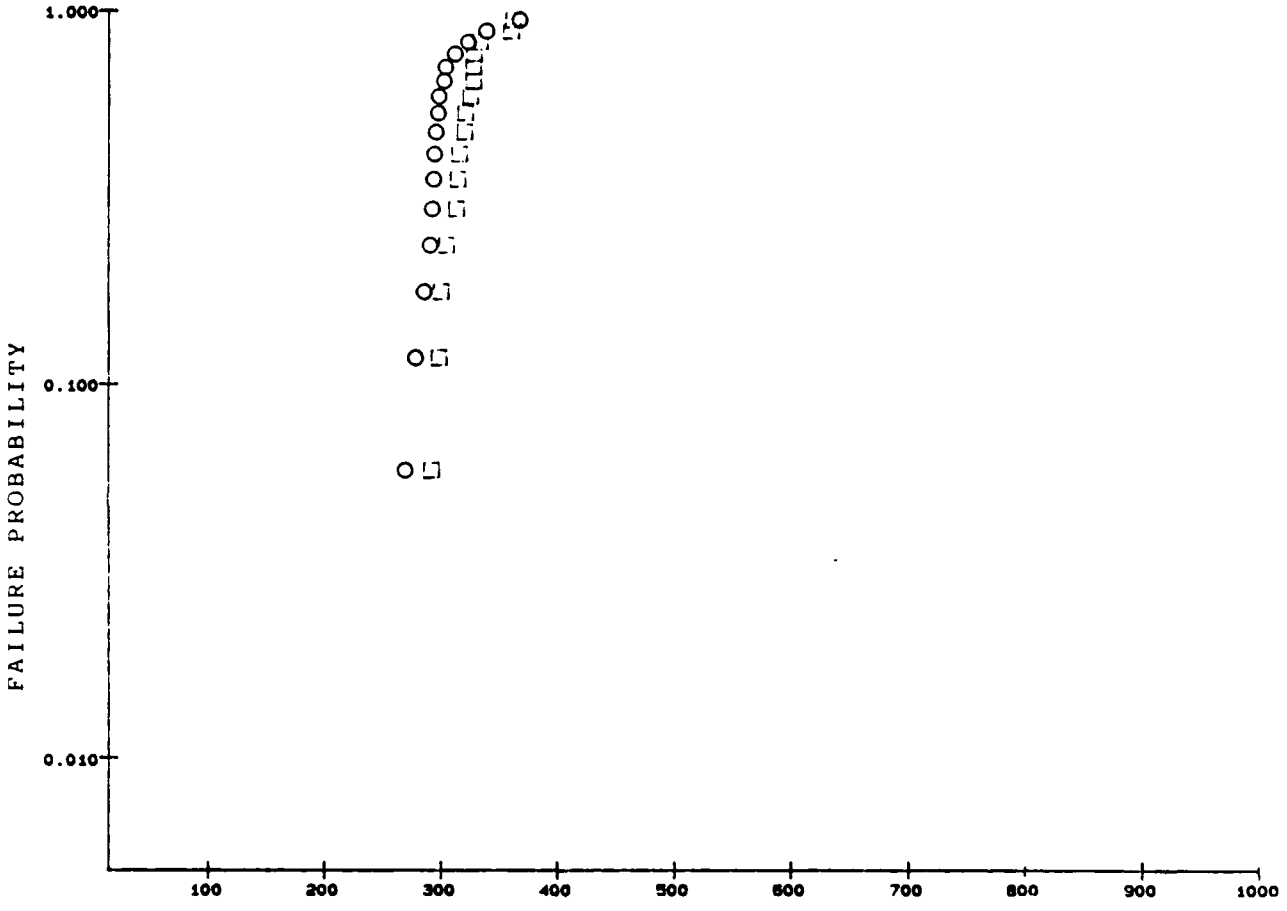

FIGURE 17.

WEIBULL PLOTS OF Si_3N_4 COATED FIBERS

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